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THE RHEOLOGICAL PROPERTIES OF FLUID DENTURE-BASE RESINS, (U)
MAR 77 S G VERMILYEA, J M POWERS, A KORAN

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The pour type or fluid resins for the fabrication of denture bases have gained in popularity over the last decade. Their chief advantage lies in the dramatic reduction in the time required for processing and finishing the final restoration. The fluid resin technique requires only 1/7 to 1/10 of the 15 to 20 hours normally necessitated by conventional compression molding methods. In view of the nearly 30,000 denture bases fabricated annually by Army technicians, such a reduction would obviously result in substantial savings of fiscal and manpower resources.		

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A common problem associated with the fluid resins is the presence of voids in the finished restoration. These voids are thought to be dependent upon the flow characteristics of the resin. These characteristics, in turn, are governed largely by rheological phenomena. Unfortunately, sufficient information relevant to the flow properties of fluid resins has been heretofore unavailable.

The present investigation determined the flow properties of several fluid denture base resins. The resultant data permits the establishment of definitive criteria for selection of materials suitable for military use. Additionally, techniques relevant to the laboratory manipulation of the fluid resins have been delineated. Dissemination of such information regarding the fluid resins will aid in the development of new and improved materials for military dentistry.

This study determined the rheological properties of six commercial fluid resins by rotational viscometric techniques. The viscosity of Hyflo (A), Pronto II (B), Pro-Fit (C), Porit (D), TruPour (E), and Pour-n-Cure (F) was measured as a function of time and rotational speed at a temperature of $23 \pm 0.05^\circ\text{C}$. A calibrated spindle was rotated at rates of 10, 20 and 50 revolutions per minute (rpm). The viscosity in centipoise (cp) was recorded for 6 min after initiation of mixing or until the viscosity of the products exceeded the range of the recorder. Data were evaluated by analysis of variance and means were compared by Scheffe's method at the 95% level of confidence. The initial viscosities at 10 rpm were: A, 67 cp; B, 104 cp; C, 133 cp; D, 133 cp; E, 267 cp; and F, 575 cp. The Scheffe' interval was 21 cp. The viscosity of all materials increased with time. At 3 min after the start of mixing the viscosities at 10 rpm were: A, 342 cp; B, 371 cp; C, 282 cp; D, 475 cp; E, 625 cp, and F, 2,933 cp. The Scheffe' interval was 129 cp. At increased rotational speeds Material F was pseudoplastic. Initially, however, behavior of the other resins was characterized by dilatancy. At 50 rpm the initial viscosities were: A, 87 cp; B, 133 cp; C, 160 cp; D, 155 cp; E, 301 cp; and F, 322 cp. The Scheffe' interval was 21 cp for materials and 11 cp for rotational speed. Changes in viscosity with increased rotational speed appear to reflect the effects of rheologic behavior and the rate of the setting reaction of each material

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THE RHEOLOGICAL PROPERTIES OF
FLUID DENTURE-BASE RESINS

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Commercial materials and equipment are identified in this report to specify the experimental procedure. Such identification does not imply official recommendation or endorsement or that the equipment and materials are necessarily the best available for the purpose.

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Synopsis

The rheological properties of six commercial fluid denture resins were determined by rotational viscometry. Initial viscosity ranged from 67 to 575 centipoise at a rotational speed of 10 revolutions per minute. All resins showed initial non-Newtonian flow behavior and increased viscosity with time.

The use of pour-type (fluid) resins for the fabrication of denture bases has increased markedly over the past decade. Advantages offered by the fluid resins include: (1) Reduction of processing time; simplification of flasking and deflasking procedures; and (3) reduction of time required for finishing and polishing the cured prostheses.¹

Previous studies on fluid resins have been addressed to measurement of physical and mechanical properties, modification and refinement of processing procedures, and assessment of the effect of investment materials on the quality of the finished restoration.²⁻³ Products made from pour-type resins often exhibit voids and other surface imperfections. It has been suggested that the defects may be the result of pouring a resin that is too viscous.⁵ Unfortunately, sufficient information regarding the viscous properties of the pour resins is not available.

The present investigation was conducted to determine the apparent viscosity of several pour-type denture base resins as a function of time and rotational speed at a constant temperature.

Materials and Methods

Six pour-type denture base resins were obtained from proprietary sources. The manufacturers and batch numbers are listed in Table 1.

The polymer and monomer components of the materials were proportioned in accordance with manufacturer's recommended powder-liquid ratios to yield mixtures of approximately 40 cc. All mixtures and measurements were made within a constant temperature chamber maintained at $23 \pm 0.05^\circ\text{C}$.

Viscosity of the test resins was determined with a rotational viscometer.* Shearing was caused by the rotation of a disc (diameter 2.12 cm) and rod (diameter 0.45 cm) spindle assembly⁺ at rates of 10, 20 and 50 revolutions per minute (rpm). Three trials were made with each material at each shear rate. The pneumatic output of the viscometer was converted to an electrical signal by a pneumatic-electric converter[#] and recorded on a y-time recorder.^Ω Data were obtained as viscosities in centipose (cp) by calibration of the spindle with a Newtonian standard.[§] Recording of the viscosities of five materials (A, B, D, E, F) was begun 45 seconds after the start of mixing. Initial measurements for one resin (C) were made 90 seconds after commencement of sample preparation. The viscosity of each material was recorded continuously for a period of six minutes following the initiation of mixing or until the viscosity of the mix exceeded the range of the recorder.

All data were evaluated statistically by analysis of variance and means compared using Scheffe's method at the 95 percent level of confidence.⁹

* Rheolog Model 1/4 RVT-RL-199, Brookfield Engineering Corp., Stoughton, MA 02074.

+ Spindle No. 5, Brookfield Engineering Corp., Stoughton, MA 02074.

Pneumatic-Electric Converter, Brookfield Engineering Corp., Stoughton, MA 02072.

Ω Model XY530TM, Esterline Angus, Unit of Esterline Corp., Indianapolis, IN 46404.

§ Viscosity Standard No. N3500, Lot No. 72025p, Cannon Instrument Co., State College, PA 16801.

Results

The viscosity of each material increased markedly with time. The changes in the viscosities of the resins with time at 10 rpm are shown in Figure 1. Initial viscosity values ranged from 67 cp for material A to 575 cp for material F. The mean values of the initial viscosity at 10 rpm of C and D were statistically equivalent at the 95 percent level of confidence (Table 2). The mean values of the viscosity of A, B, E, and F were statistically different from each other and from those of the other materials at this rotational speed. The Scheffe' interval was 21 cp.

At three minutes (materials A, B, D, E, and F) and 3 1/2 minutes (material C), after the start of mixing (Table 2), the viscosity of F had increased to 2,933 cp. The viscosities of the other materials had increased to values only slightly above (625 cp, E) or markedly below (283 cp, C) the initial viscosity of F (575 cp). The mean values of the viscosity of A, B, and C were statistically equivalent at the 95 percent level of confidence. The viscosities of B and D were also equivalent at this confidence level. The Scheffe' interval was 129 cp.

The effect of rotational speed on the initial viscosity of the resins is shown in Figure 2. The viscosity at a rotational speed of 50 rpm ranged from 0.6 times (F) to 1.3 times (A) the viscosity measured at 10 rpm. Materials A, B, C, D, and E demonstrated significant increases in viscosity with increased rotational speeds indicating dilatant flow behavior. Material F exhibited pseudoplasticity or shear thinning over the range of rotational speeds employed. The Scheffe'

interval was 11 cp. As the time from the start of mixing increased, the effect of rotational speed on the viscosity of all but one of the resins (F), decreased. The changes in viscosity with rotational speed at three minutes after the start of mixing are shown in Figure 3. At this time, the viscosity at 50 rpm ranged from 0.6 times (F) to 1.03 times (B) the viscosity measured at 10 rpm. Materials A, B, C, and D behaved as Newtonian liquids while materials E and F exhibited pseudoplastic flow. The Scheffe' interval was 66 cp.

Discussion

The viscosity of a polymerizing monomer-polymer mixture is affected by many factors. The particle size, molecular weight, and initiator content of the polymer as well as the molecular weight, and presence of cross-linking agents, inhibitor and accelerator in the monomer influence the rate of polymerization and contribute to the viscous properties of the mixed resins.

The statistical differences between the initial viscosities of some of the resins may reflect these compositional variations but possess little practical significance. A range of viscosities for the optimal clinical use of the pour resins has not been established. Though the initial viscosity of F is nearly nine times that of A, it is probably still low enough to allow the resin to be poured into the prepared mold.

The viscosity of the mixed resin as the time from the start of mixing increases is a much more important factor in the clinical handling of these materials. A resin possessing a low viscosity for

a suitable period of time would be convenient where multiple molds were to be poured from a single mix. The lower values of viscosity for A, B, C, D, and E at three minutes from the start of mixing suggest that a longer working time may be available with these materials than with F.

The effect of rotational speed on the viscosity of the mixed resins may be due to the combined effects of rheological behavior and reaction rate. The mixed materials are essentially powder-liquid slurries. At low rotational speeds, the powder particles may be able to slide past one another as the liquid acts as an interstitial lubricant. As the rotational speed increases, the powder particles become packed together with no intervening liquid. The force required to maintain a constant rotational speed increases and a higher viscosity is recorded at these increased rates of shear.

Additionally, the continued stirring action of the rotating spindle may increase the rate of polymerization. At higher rotational speeds the interaction of the monomer with the accelerator and other monomer molecules may be enhanced. The resultant accelerated reaction rate results in an increase in viscosity. Thus, the combined effects of powder particle interaction and accelerated polymerization may produce the shear thickening or dilatant flow observed experimentally with materials, A, B, C, D, and E.

As the time from the start of mixing increases, the continued mixing action of the spindle may interfere with the formation of the polymer chains. Once the rate of polymerization has reached a maximum, continued mixing will no longer accelerate the reaction, but instead

reduce the viscosity of the mix. Thus, the phenomena of powder particle interaction and reaction rate become antagonistic and the magnitude of the shear rate effect is reduced. The pseudoplasticity observed initially with material F and at 3 minutes with materials E and F may reflect this antagonism. A balance between the particle interaction-reaction rate phenomena and the interference with the developing polymer chains may result in the Newtonian behavior observed with materials A, B, C, and D.

Clinically, the initial dilatant behavior of the fluid resins A, B, C, D, and E suggests that the introduction of the mixed resin into the mold should be accomplished slowly. Rapid pouring or injection of these materials may create high shear rates and increase the viscosity such that flow into the fine details of the mold is prohibited.

The pseudoplasticity observed with material F indicates that rapid pouring of the resin may enhance the flow into the fine details of the mold. However, high shear rates which result in apparent pseudoplastic behavior may adversely affect the mechanical properties of the cured resin. Further research into the effects of shear rate on the detail reproduction of the fluid resins and on their mechanical properties would be invaluable in establishing guidelines for the clinical handling of these materials.

Conclusions

The rheological behavior of six pour-type denture base resins was studied by rotational viscometric techniques.

Considerable variation existed in the initial viscosities of the materials evaluated. The values of the initial viscosities ranged from 67 to 575 cp at a rotational speed of 10 revolutions per minute.

The initial viscosity of all but one of the materials (F) increased significantly with increases in rotational speed, suggesting dilatant flow behavior.

The viscosity of each material increased markedly with time. The viscosity of material F increased at a faster rate than did those of the other materials.

Early introduction of the mixed materials into the prepared mold may help minimize the presence of voids and the loss of fine detail in the final restoration.

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TABLE 1: Materials Evaluated in the Study

Code	Material	Batch Number Powder	Batch Number Liquid	Powder/Liquid Ratio by Volume	Manufacturer
A	Hyflo	061776	061776	2.5/1	Hygenic Dental Mfg. Co. Akron, OH 44310
B	Pronto II	249106	069204	2.5/1	Vernon-Benshoff Co. Albany, NY 12201
C	Pro-Fit	084040	104013	2.5/1	Howmedica, Inc. Chicago, IL 60632
D	Porit	P0372J	M0475C	2.35/1	Product Research Laboratory Cambridge, MA 02130
E	TruPour	74226	74275	2.15/1	Dentsply International, Inc. York, PA 17404
F	Pour-n-Cure	040476	020176	2/1	Coe Laboratories, Inc. Chicago, IL 60658

TABLE 2: Viscosity of the Fluid Resins^{*}

Material	Initial Viscosity (cp)	Viscosity at 3 Minutes After the Start of Mixing (cp)
A	67 (14) ⁺	342 (29)
B	104 (7)	371 (8)
C	133 (14)	283 (38) [#]
D	133 (14)	475 (25)
E	267 (29)	625 (43)
F	575 (0)	2,933 (260)

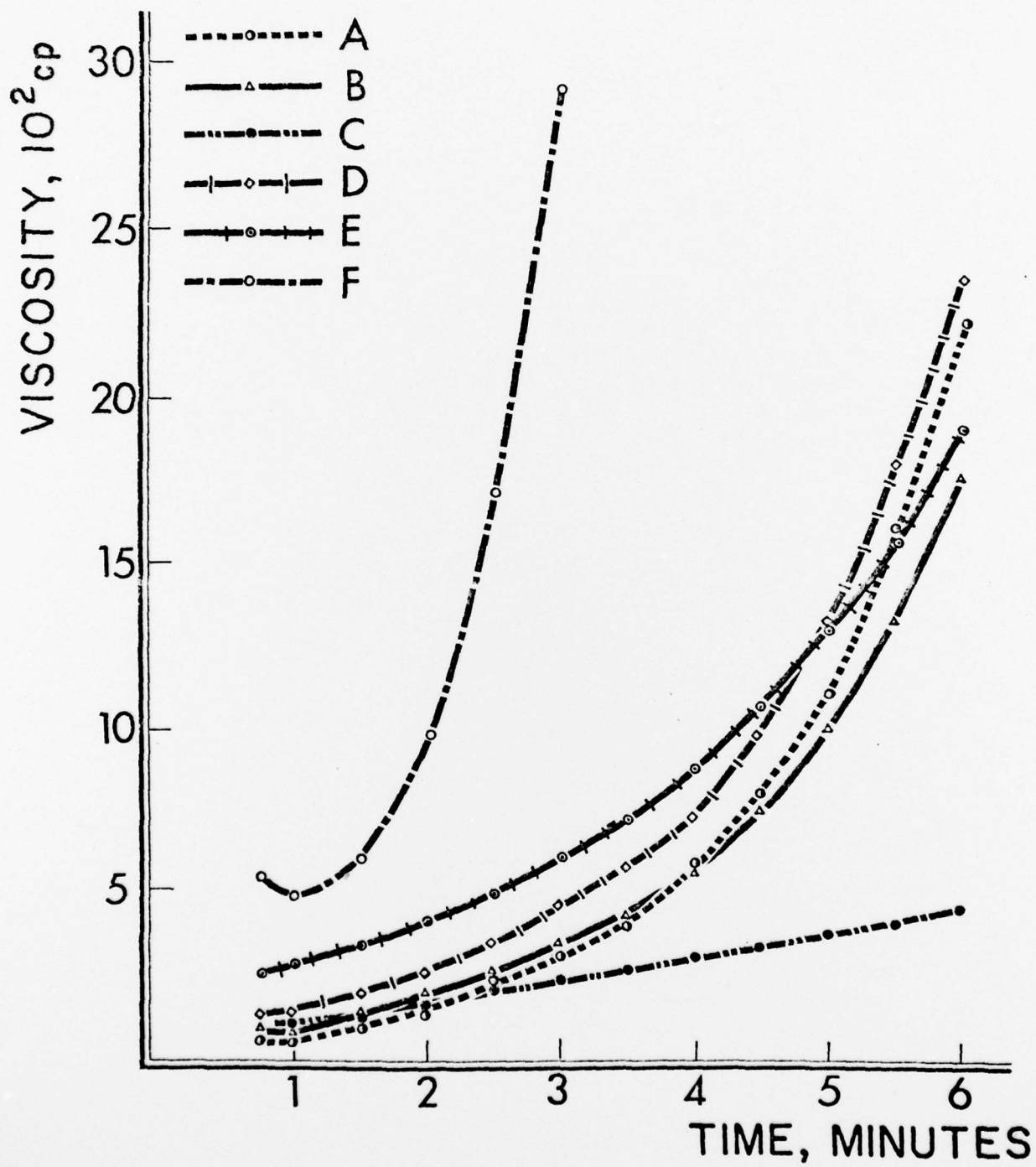
* The viscosities were obtained at a rotational speed of 10 revolutions per minute.

+ Means with standard deviations in parentheses were determined from three replications. Scheffe' intervals were computed to be 21 cp for the initial viscosities and 129 cp for the viscosities at three minutes from the start of mixing.

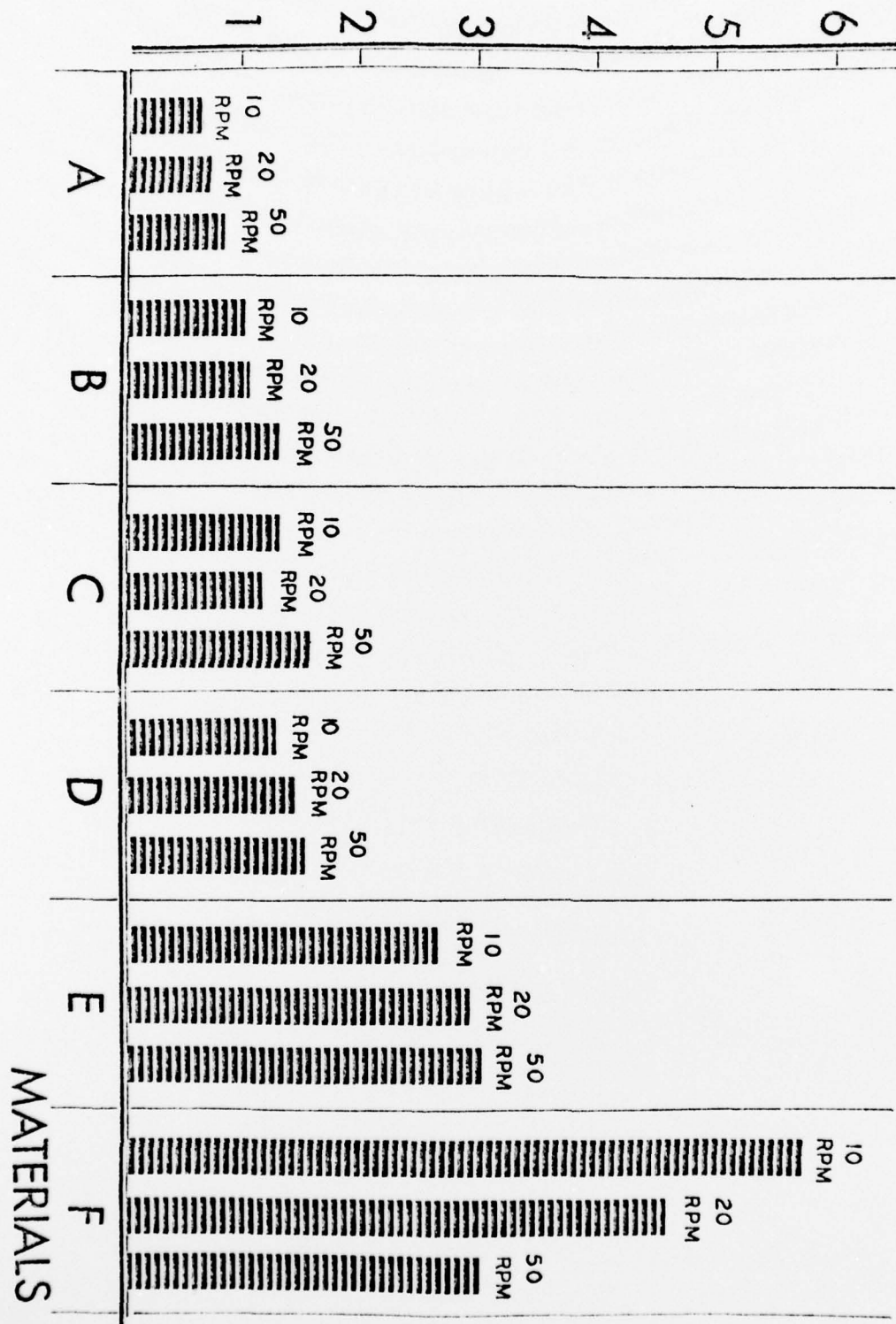
Viscosity (cp) at three and one-half minutes after start of mixing.

Legends for Figures

- Figure 1. The Change in Viscosity with Time at 10 rpm.
- Figure 2. The Initial Viscosity of Each Material as a Function of Rotational Speed.
- Figure 3. The Viscosity of Each Material at Three Minutes after the Beginning of Mixing as a Function of Rotational Speed.



VISCOSITY, 10^2cp



VISCOSITY, 10^2cp

30
25
20
15
10
5

A

10 RPM
20 RPM
50 RPM

B

10 RPM
20 RPM
50 RPM

C

10 RPM
20 RPM
50 RPM

D

10 RPM
20 RPM
50 RPM

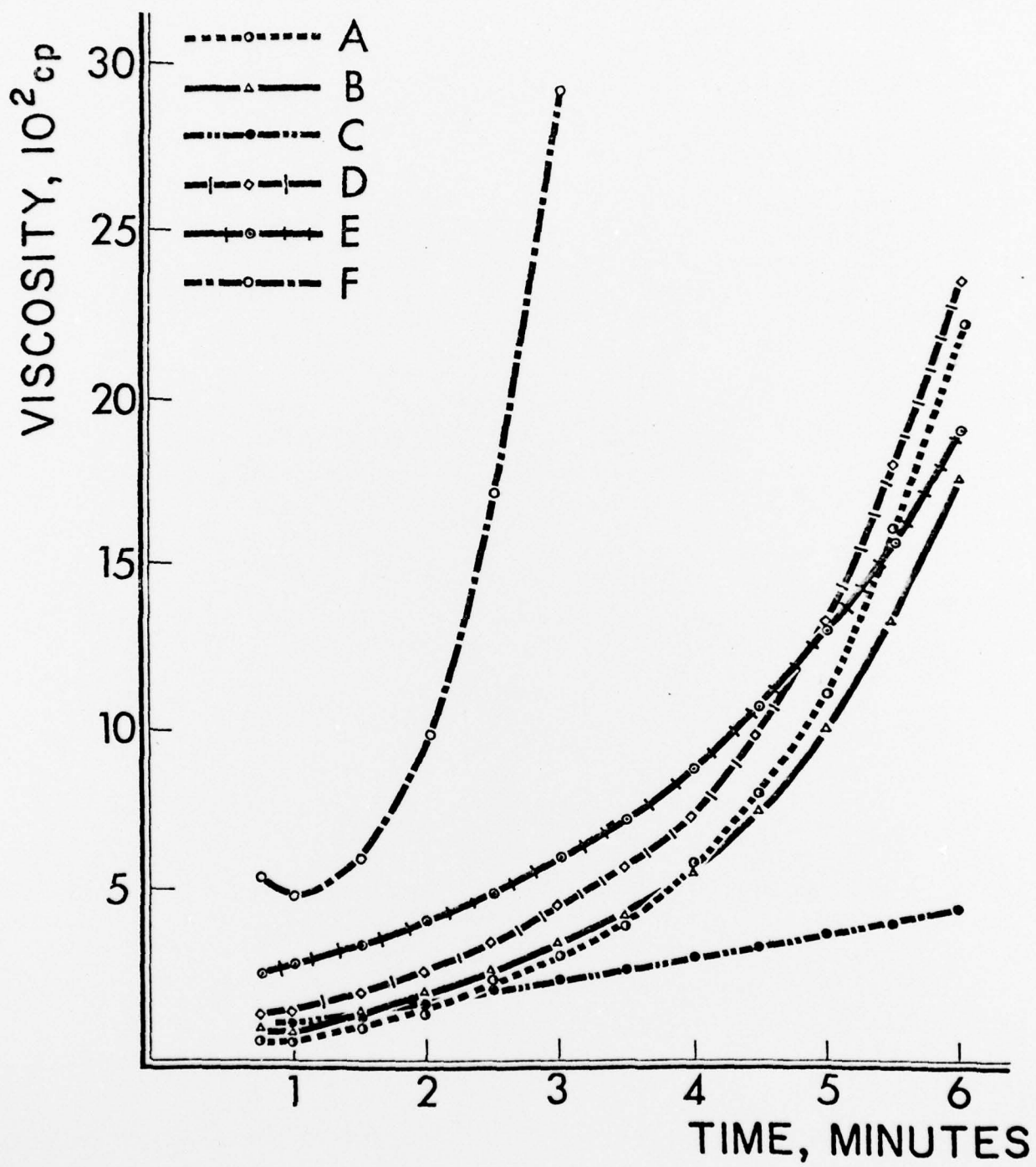
E

10 RPM
20 RPM
50 RPM

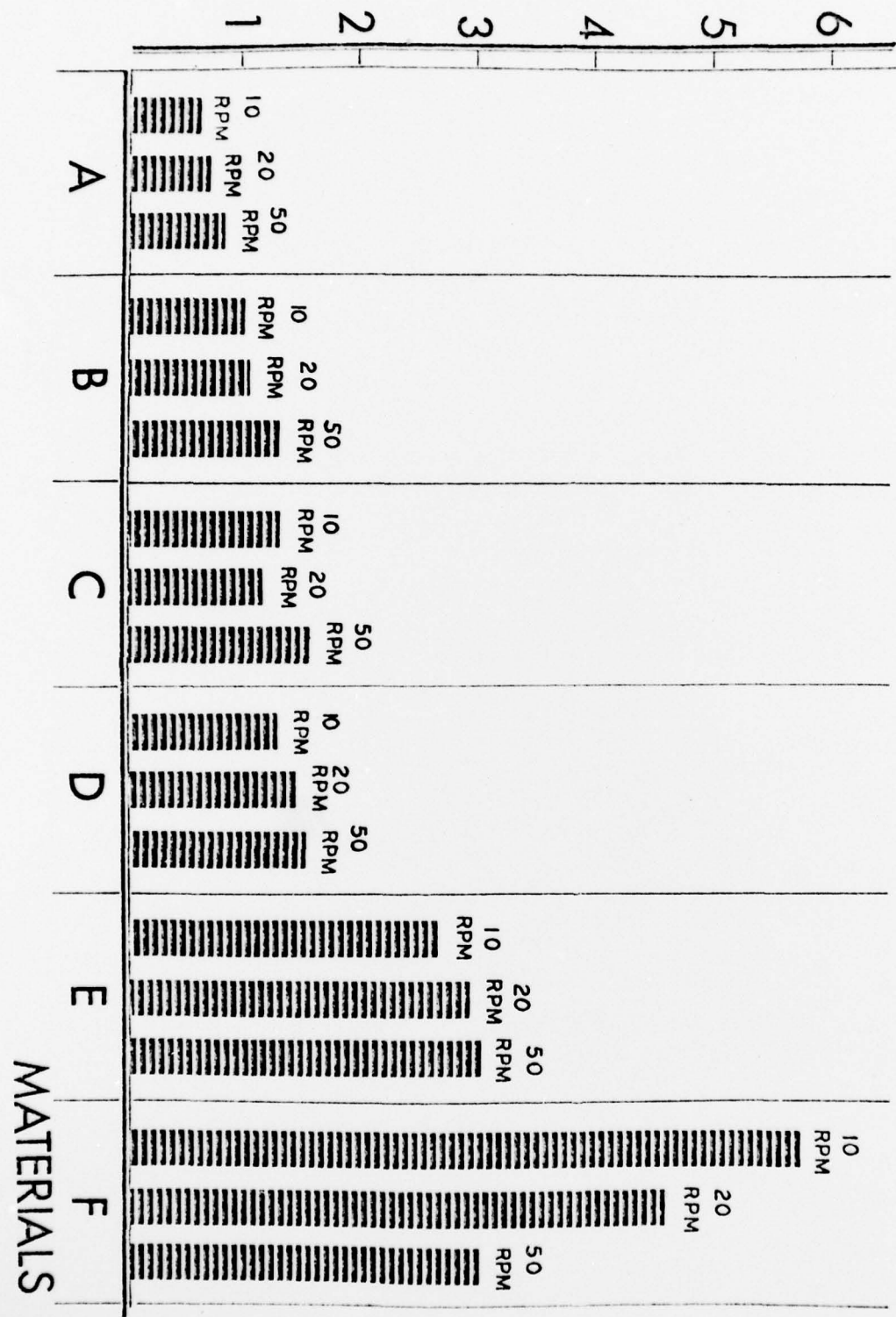
F

10 RPM
20 RPM
50 RPM

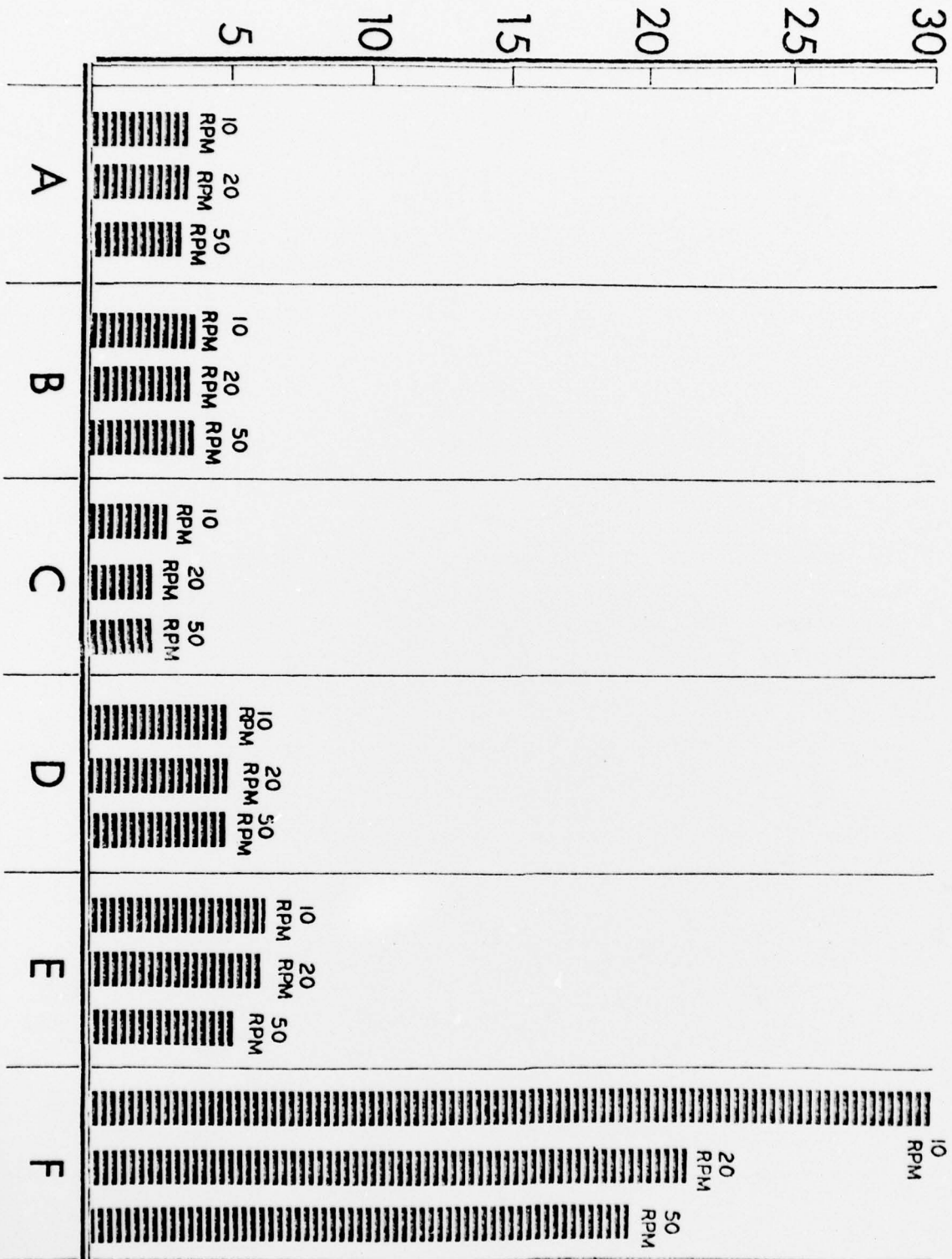
MATERIALS



VISCOSITY, 10^2cp



VISCOSITY, 10^2cp



MATERIALS